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Journal of Prosthodontic Research 55 (2011) 146–153
Journal of Prosthodontic Research
www.elsevier.com/locate/jpor

Original article

The effect of surface treatment on bond strength of layering porcelain and hybrid composite bonded to zirconium dioxide ceramics

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Received 22 April 2010; received in revised form 20 October 2010; accepted 27 October 2010
Available online 26 November 2010

Abstract

Purpose: The purpose of this study was to investigate the differences between Rocatec (as surface treatment) and #600 polishing (as control) on shear bond strength of layering porcelain and hybrid composite to zirconium dioxide ceramics.
Methods: Manufactured zirconia blocks used in this study were yttrium partially stabilized zirconia (YTZ®), and veneering materials were NobelRondo Zirconia Dentin A2 High Value (NZR) and Estenia C&B (ES). Total 48 zirconia blocks were fabricated (10 mm × 10 mm × 20 mm). The blocks of 24 each were treated by Rocatec and #600 paper, respectively. Surface treated zirconia blocks were divided into two groups, according to veneering materials of NZR and ES. NZR was fired and ES was polymerized to zirconia. The fabricated specimen was fixed to mounting jig and applied shear force using the universal testing machine at a crosshead speed of 0.5 mm/min. All results were statistically analyzed by two-way ANOVA and Tukey's test. EPMA analysis and SPM analysis of specimen interface were carried out.
Results: Mean shear bond strength of each condition was: NZR/#600; 23.3 (S.D. ±7.0) MPa, NZR/Rocatec; 26.9 (S.D. ±7.0) MPa, ES/#600; 10.7 (S.D. ± 2.4) MPa, ES/Rocatec; 12.5 (S.D. ± 0.8) MPa.
Conclusions: From the results of this study, shear bond strength of layering porcelain to zirconia was higher than that of restorative hybrid resin. However the more study will be needed, the appropriate choice of materials became the gides to the expansion of the applied cases of metal-free prothesis.

Keywords: Bond strength; Zirconium dioxide ceramics; Layering porcelain; Hybrid composite; Surface treatment

1. Introduction

Zirconium dioxide ceramics (zirconia), one of the high strength ceramics is a biomaterial that has excellent strength and toughness [1–4]. In dentistry, this has been used as framework material of all-ceramic restorations and esthetic dentistry. And now, it is possible to fabricate from one crown to six units fixed partial dentures [5,6]. And these were highlighted as materials that have good function and strength as same as metal framework. However, on the other hand, some clinical cases of minor problem such as chipping of veneering porcelain from the surface of framework [6–9] were reported. Bonding of hybrid resin to zirconia also has problems. Because it is usually considered that the adhesion between the different kinds of materials such as resin and ceramics is weak. In this study, we have tested on the assumption that we use the hybrid resin as veneering materials for zirconia based crown. The consideration of the possibility of applying it as veneering materials was needed. Therefore, for restorative veneering materials, it is necessary to bond strongly and prevent from crack or delamination from zirconia surface. In many studies, bond strength of porcelain [10–12] and hybrid resin [13–15] to metal frameworks was reported in detail. Furthermore, for surface treatment, Rocatec system has been used. Rocatec is the tribochemical method for silicatising surfaces. This system can create a bond between the dental materials, not only metal alloy

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1883-1958
doi:10.1016/j.jpor.2010.10.005

Table 1
Materials used in this study.

Materials	Code	Lot no.	Manufacturer	Main components
Layering porcelain				
NobelRondo Zirconia (Dentin A2 High Value)	NZR	0305	Nobel Biocare, Gothenburg, Sweden	SiO_2, Al_2O_3, Na_2O, K_2O
Restorative hybrid resin				
Estenia C&B	ES	0021AA	Kuraray Medical, Tokyo, Japan	UTMA, 92 wt% micro filler
Priming agents				
Espe Sil	SI	1915	3M Espe, St. Paul, USA	Silane in ethanol
Bonding agent				
S3 Bond	–	011116	Kuraray Medical, Tokyo, Japan	Bis-GMA, MDP, HEMA

UTMA: Urethane tetramethacrylate, Bis-GMA: Bisphenol A diglycidylmethacrylate, MDP: Methacryloyloxydecyl dihydrogen phosphate, HEMA: Hydroxy ethyl methacrylate.

[16,17], but also alumina and zirconia ceramics [18–20]. Now, the bond strength of resin luting cement [21,22] to zirconia was tested, but hybrid composite resin was not.

We consider if it is possible to establish the system for secure bond force of veneering materials to zirconia framework, it will become a guide for choice of safety and reliable treatment method. To use different restorative materials according to the cases will be widely applicable for the use of metal-free restorations. Moreover, technical development of CAD/CAM system will be able to expect the high quality of prosthetic restoration, standardization of fabrication accuracy, simplicity of manufacturing process, and improvement of surroundings of laboratory works. From these reasons, it is necessary to evaluate the effect of firing and bonding method for layering porcelain and hybrid resin materials to the surface of zirconia frameworks. And now, bond strength of dental ceramics is evaluated using different kinds of method [23,24]. Therefore, it is difficult to compare and assess the results from each test, so evaluation and comparison from standardized method is important.

The purpose of this study is to evaluate the bond strength of layering porcelain and restorative hybrid resin to zirconia and investigated the effects of different surface treatment.

2. Materials and methods

Manufactured zirconia blocks used in this study were 5%-yttrium dioxide partially stabilized zirconia (YTZ®, Nikkato, Tokyo, Japan). The composition is 5.03 wt% Y_2O_3–94.67 wt% ZrO_2. Materials used in this study are shown in Table 1. NobelRondo Zirconia Dentin A2 High Value as layering porcelain (NZR, Nobel Biocare, Gothenborg, Sweden), Estenia C&B as hybrid resin (ES, 92 wt% micro filler, Kuraray Medical, Tokyo, Japan), Espe Sil as silane coupling agent (3M Espe, St. Paul, USA) and S3 Bond as bonding agent (Kuraray Medical, Tokyo, Japan) were selected.

This study is carried out by two-way factorial experiment. Factor A is two kinds of restorative materials (NZR and ES), and Factor B is also two kinds of surface treatment methods (#600 and Rocatec).

Total 48 zirconia blocks were fabricated in the size of 10 mm × 10 mm × 20 mm. The surface of all blocks were polished by #600 silicon carbide paper to one direction, and then ultrasonically cleaned by acetone and distilled water for 15 min, respectively. The fabricated zirconia blocks have specular surfaces. In our previous study, we used zirconia blocks polished by #600 paper as the standard surface. Also we can see visually lusterless surfaces after polishing. Half numbers (24) of blocks were tribochemically treated by Rocatec junior (Rocatec, 3M Espe, St. Paul, USA), which is blasted with 110 μm silica modified aluminum oxide particles (Rocatec Plus, 3M Espe, St. Paul, USA) for 13 s, at a pressure of 0.28 MPa, from 10 mm distance. The rest half (24) numbers of blocks were just used to this study. Rocatec and #600 treated zirconia blocks were then divided into two groups, according to veneering materials of NZR and ES, respectively.

Table 2
Firing schedule of NZR.

		NZR	
		1st Firing	2nd Firing
Drying	Preheating temperature	575 °C	575 °C
	Drying time	8 min	8 min
Firing	Heating rate	55 °C/min	45 °C/min
	Firing temperature	910 °C	900 °C
	Holding time	1 min	1 min
	Vacuum	50 hPa	50 hPa

Surface treated specimens were divided into two groups (#600 and Rocatec, $n = 12$). Each specimen was fixed to build-up mold (Japan Mecc, Tokyo, Japan) which has hole in diameter of 6 mm and 2 mm thickness. Layering porcelain was built up with exclusive liquid (NobelRondo Build-up Liquid, NobelBiocare, Gothenborg, Sweden), and then condensed. After condensation, specimen was removed from the mold. The specimens were fired in the porcelain furnace (AUSTROMAT D4, DEKEMA, Freilassing, Germany) according to the manufacturer's instructions. The firing schedule of NZR is shown in Table 2. Due to the porcelain shrinkage, a total of two separate firings were required to make the correct diameter and thickness.

The bonding method was shown in Table 3. The surface of #600 was treated by bonding agent (S3Bond, Kuraray Medical, Tokyo, Japan), and that of Rocatec was treated by silane coupling agent (SI) prior to bonding agent. Then plastic ring (6 mm in diameter, 2 mm thickness) was placed on the surface

Table 3
Polymerization and adhesion methods of hybrid resin to zirconia surface.

Materials	Treatment method
Espe Sil	Apply SI → dry for 5 min
S3 Bond	Apply bonding agent → leave for 20 s → air blow → light cure for 10 s
Estenia C&B	Light cure for 60 s → heat cure 110 °C for 15 min

treated area, and filled with ES and then pressed with glass plate. Specimen was polymerized using heat and light cure unit (Twin Cure, Shofu, Kyoto, Japan) while pressing. After polymerization, plastic ring was cut and then put into distilled water and stored in incubator keeping at the temperature of 37 °C for 24 h.

Adhesive condition of laboratory was 23 ± 2 °C and relative humidity of 50 ± 5%.

The specimen was fixed to mounting jig and applied shear force using the universal testing machine (Servo Pulser EHF-FD1, Shimadzu, Kyoto, Japan). Load was applied at a crosshead speed of 0.5 mm/min. Shear bond strength in MPa was calculated. Mounting jig and schematic images of shear bond test were shown in Fig. 1.

All recorded results were statistically analyzed using two-way analysis of variance test (ANOVA). And Tukey multiple comparisons test was also used to assess the differences among the materials. This test was performed to $n = 12$ specimens/group.

Zirconia specimens used in shear bond test were cut by diamond disc, and embedded into epoxy resin. Fractured surfaces of zirconia specimens were analyzed using EPMA (S-400, Hitachi, Tokyo, Japan) with an acceleration voltage of 10 kV, 200 magnification about Al, Zr, Y, K, Si for layering porcelain and Al, Zr, Y, Si for hybrid resin, respectively.

Bonding surfaces of specimens (#600 and Rocatec) used in shear bond test of layering porcelain to Zirconia were analyzed by scanning probe microscope (SPM, Shimadzu, Kyoto, Japan).

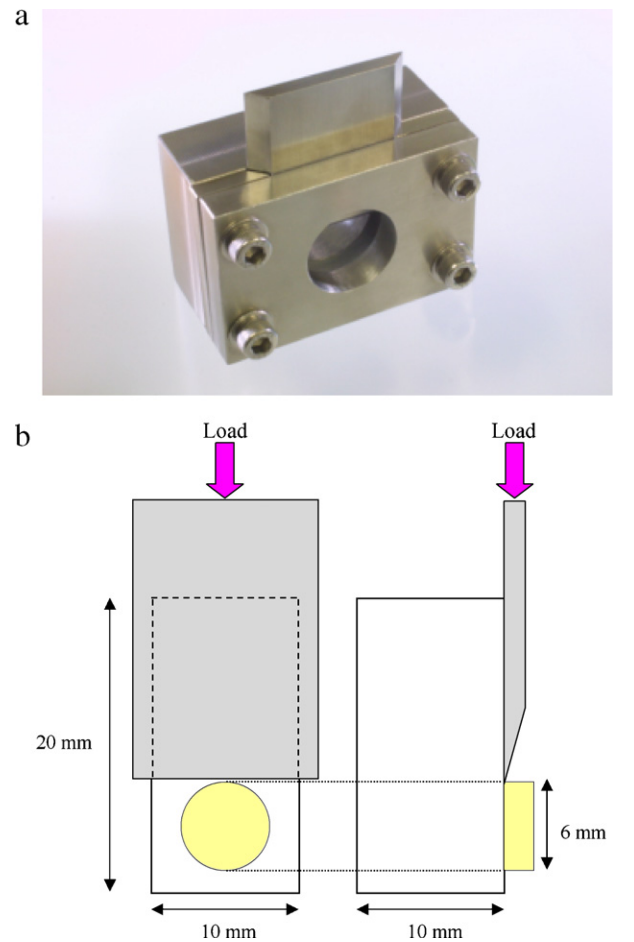

Fig. 1. Shear test device (a) and schematic image of shear bond test (b: left: front view; right: lateral view).

Table 4
Results of two-way ANOVA.

Source	s.s.	d.f.	m.s.	Fo
A: Restorative material	2187.67	1	2187.67	37.83**
B: Surface treatment	9.89	1	9.89	1.78
A × B	89.02	1	89.02	0.19
e	1131.54	44	25.72	
T	3418.13	47		

s.s.: Sum of Squares, d.f.: Degree of freedom, m.s.: Mean square.
** $p < 0.01$.

3. Results

3.1. Shear bond strength between veneering materials to zirconia

The result of two-way ANOVA was shown in Table 4. Factor A was higher significant difference, but factor B and A × B had no significant differences. Mean shear bond strength of the difference of veneering materials (ES and NZR) was shown in Fig. 2, and that of surface treatment was shown in Fig. 3. Mean shear bond strength of each condition was: NZR/#600; 23.3 (S.D. ±7.0) MPa, NZR/Rocatec; 26.9 (S.D. ±7.0) MPa, ES/#600; 10.7 (S.D. ±2.4) MPa, ES/Rocatec; 12.5 (S.D. ±0.8) MPa.

3.2. EPMA analysis of interfacial surface

EPMA photographs of NZR and ES to zirconia were shown in Figs. 4 and 5. The surfaces of Rocatec treated specimens were scraped, and observed the aspects that alumina particle attached in the scraped surface more than #600 treated surfaces. Furthermore for NZR specimens, most of yttrium was contained in zirconia, and K, Al and Si were in NZR. For interfacial surface of ES specimens, more alumina layer was observed on Rocatec surface than #600 treated surfaces. And ES has high contents of alumina and silica, so they were observed much more than other components of zirconia and yttrium.

3.3. SPM (scanning probe microscope) analysis

Table 5 shows the fracture mode of surface divided by numbers of specimen. Cohesive fracture and cohesive/adhesive

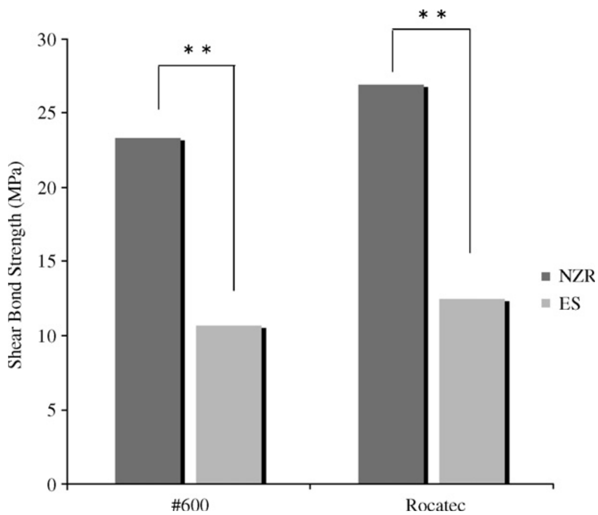

Fig. 2. Mean shear bond strength of materials and surface treatment ($^{**}p < 0.01$).

fracture were observed to NZR of zirconia surface, and surface fracture was observed only to ES. Fig. 5 shows SPM photographs of NZR and zirconia recorded maximum shear bond strength. For fracture surface treated with Rocatec, surface layer of NZR was comparatively smooth, whereas surface layer of zirconia showed roughness. Cohesive fracture was observed in NZR attached to zirconia surface.

4. Discussion

Zirconia ceramics is oxidized material of metal zirconium. It has high strength and high toughness, and classified into three kinds of pure-zirconia, stabilized zirconia, and partially stabilized zirconia. Recently, zirconia applying in daily practice of dentistry was partially stabilized zirconia. This was called tetragonal zirconia polycrystal (TZP), and by means of addition of yttrium, that has become the most stabilized states as a high strength and high toughness material. In this study, yttrium oxide partially stabilized zirconia applied in clinical dentistry was selected.

Layering porcelain means the porcelain that is built up and fired onto the framework. This was taken account of differences of thermal expansion ratio to frameworks, mechanical properties and so on. Therefore layering porcelain is manufactured as different materials with porcelain of metal–ceramic restoration. Layering porcelain NZR was especially for zirconia framework, and its main compositions were SiO_2 and Al_2O_3. In addition, NaO_2 and K_2O were also included a small percentage. When we build up the veneering materials onto the zirconia or metal framework, we have sometimes used the opaque porcelain to cover or adjust the color of framework material. However, the delamination or cluck of veneering porcelain from the metal framework of porcelain fused metal crown were recognized to start from opaque layer, and according to the report from that there were no significant differences between using an opaque porcelain or not and components of opaque and dentin materials [11], therefore we selected NZR, layering porcelain, to build up onto the zirconia framework directly without opaque material.

Hybrid composite is used not only for inlay [25] but crown [26] or fixed partial denture [27–29]. The main component of matrix resin of ES is UTMA. This has a high content of filler and higher strength than the current composite. ES is completely polymerized by light and heat curing, and according to arrangement and improvement of the particles of filler, it keeps the abrasion by the bite force to occlusal resin material and natural teeth minimum. So, we selected ES in this study. The bonding agent is necessary for composite resin bonding. This S3 bond is the one-step bonding agent which contains Bis-GMA, MDP and HEMA. Basically, the bonding agent is a product for hard tissues such as enamel and dentin. For ceramic surface, it is considered that applying the bonding agent with silane coupling agent will cause the secure chemical bonding between the zirconia surface and hybrid composite resin. Therefore, we used this bonding agent to the zirconia surface.

As for the shear bond test on the dental materials, it is standardized from fabrication of specimens to test methods in details. Now various bonding tests were applied to many kinds of dental materials. But the studies of bonding test for metal flamework and porcelain in porcelain fused metal crown were evaluated using shear bond test [10,12] or three point bending test [11,24]. Same as the bonding test of zirconia and layering porcelain [30–33], the kind of testing method is not standardized. Also it is difficult to compare and evaluate the results recorded from various test methods, because the data were sometimes scattered. Al-Dohan et al. [30] showed it is effective to use three points bending test for bond strength of metal and porcelain, but considered to be doubtful for layering porcelain and zirconia bonding because zirconia is brittleness materials. And more consideration is needed to be able to use the same method to metal/ceramics and zirconia/porcelain bonding. In this study, we selected the shear bond test consider for fabrication of specimens, test method, plasticity of the result and brittleness of zirconia itself. And it was reported that shear bond test is the method that standard deviation and a variation coefficient are minimum and stable, so that's also the reason we selected shear bond test.

It is considered that it's possible to use clinically that the bond strength of layering porcelain to zirconia has the same or higher bond strength to metal and porcelain. Now metal framework materials were mainly gold alloy, titanium, or gold–titanium alloy. The reports by Bondioli et al. [12] and Prado et al. [10] showed that shear bond strength of metal framework and porcelain were from 32.93 MPa to 34.03 MPa. On the other hand, Al-Dohan et al. [30] reported shear bond strengths of layering porcelain to zirconia are 28.03 MPa in Procera AllZircon to Cerabien CZR and DC-Zircon to Vita D was 27.90 MPa, Dunber et al. [32] were from 23.0 MPa to 41.0 MPa, respectively. Both results were evaluated that there were no significant difference in metal/ceramic combination. In this study, for bond strength of NZR to #600 was 23.3 ± 7.0 MPa and Rocatec was 26.9 ± 7.0 MPa. It was expected that higher bond strength will be recorded on the bond test of zirconia and layering porcelain, as the surface roughness

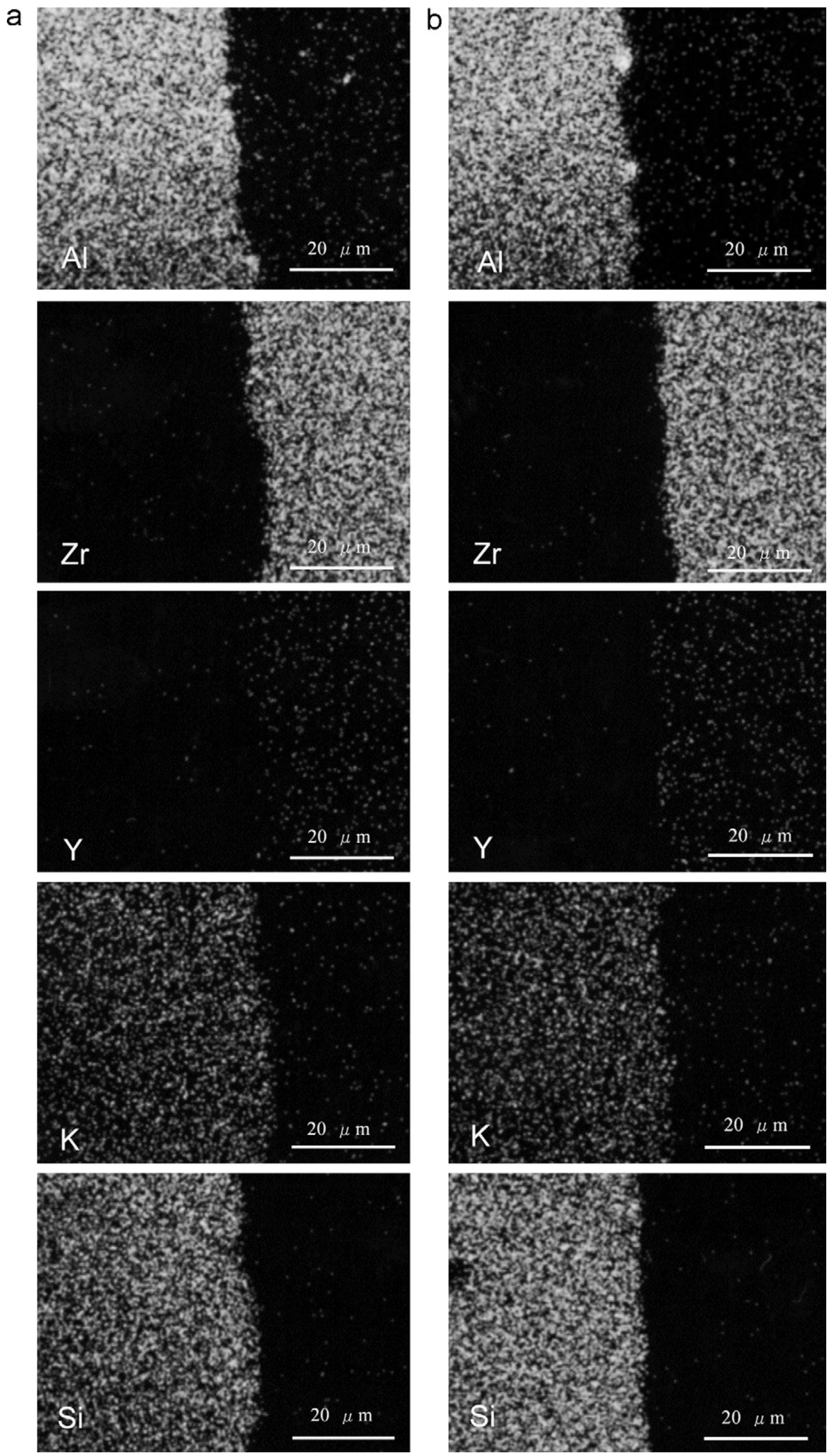

Fig. 3. EPMA photographs of layering porcelain and zirconia (a: #600 treated surface, b: Rocatec treated surface).

by Rocatec treatment shows the effect of mechanical interlocking. But there were no significant differences between the surface treatment method of #600 and Rocatec. And there are reports that the glass layer near the zirconia surface causes the chemical changes in sintering process, or the physiological change such as thermal expansion is effective to adhesion [34,35], but they were not investigated completely. Therefore it was suggested that more studies were needed that we considered the adhesion of layering porcelain to zirconia as a chemical bonding.

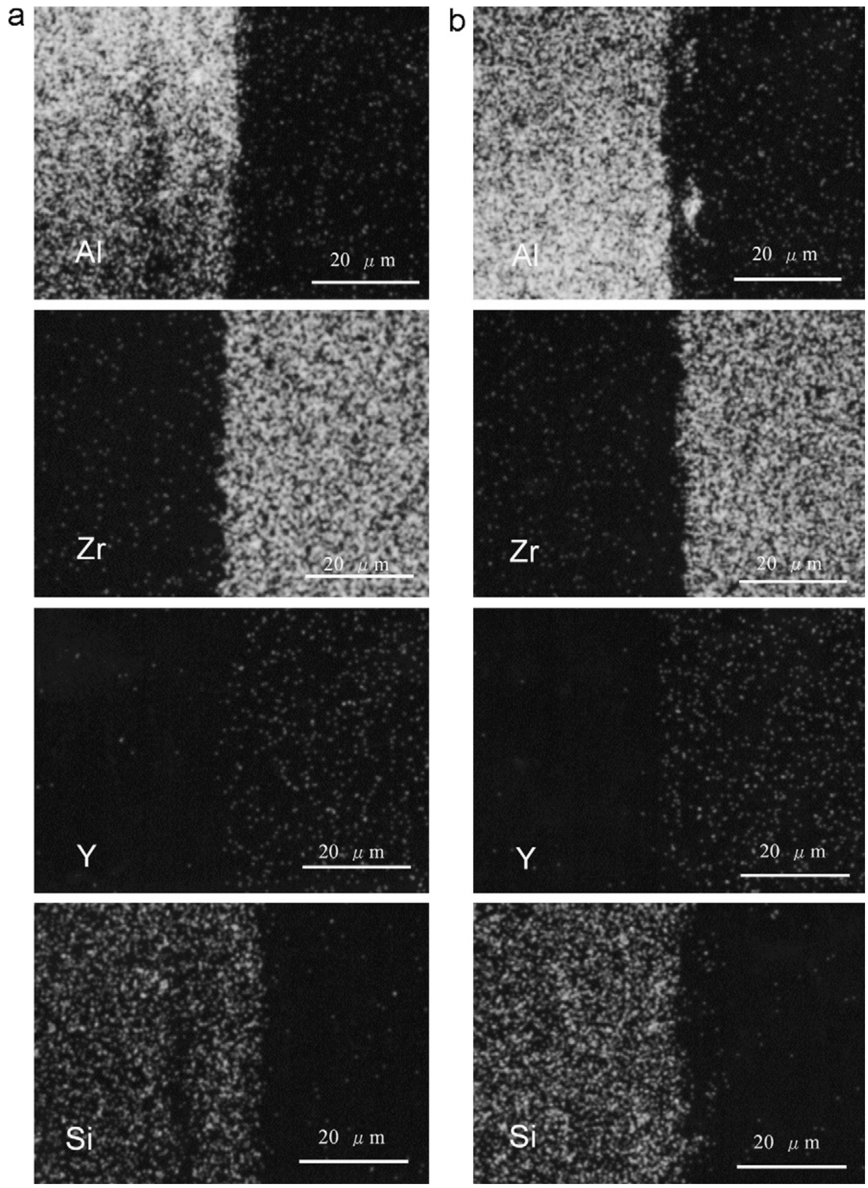

Fig. 4. EPMA photographs of restorative resin and zirconia (a: #600 treated surface, b: Rocatec treated surface).

In clinical dentistry, veneering material on zirconia framework is layering porcelain. About the adhesion of hybrid resin to zirconia, there were some reports related to repair system for delamination of veneering materials [36,37]. But there were almost no studies assumed the hybrid resin as full-covering veneering material. This is a cause that the system of resin bonding to zirconia is not cleared.

From the results of this study, adhesion of ES to zirconia is caused using bonding agent, and bond strength was lower than layering porcelain. About resin materials used in dentistry, it is reported that micro leakage will occur between the metal framework and veneering resin by water absorption [38]. When building up the resin to metal framework, it must be treated the metal surface completely [38]. This surface treatment will keep the leakage at a minimum. Furthermore, we considered that making the roughness surface brings mechanical interlocking to prosthesis and improvement of survival rate.

For the reports about shear bond strength of hybrid composite material to metal framework, Matsumura et al. [39] showed it is from 15.1 MPa to 27.8 MPa and Petridis et al. [14] showed from 17.1 MPa to 29.0 MPa. Bond strength of ES to zirconia in this study was 10.7 ± 2.4 MPa in #600 and 12.5 MPa in Rocatec. ANOVA showed there were no significant differences between the surface treatments. At present, when using hybrid composite as veneering material, it is considered that adequate silane coupling treatment and bonding will be needed.

All ceramic restoration using zirconia framework has been starting from the demands of high esthetics, and the number of cases are also increasing. In order to correspond to various clinical cases, it is important to careful consideration of properties of zirconia.

As problems of restorations using veneering materials and zirconia frameworks, chipping, delamination, crack, second

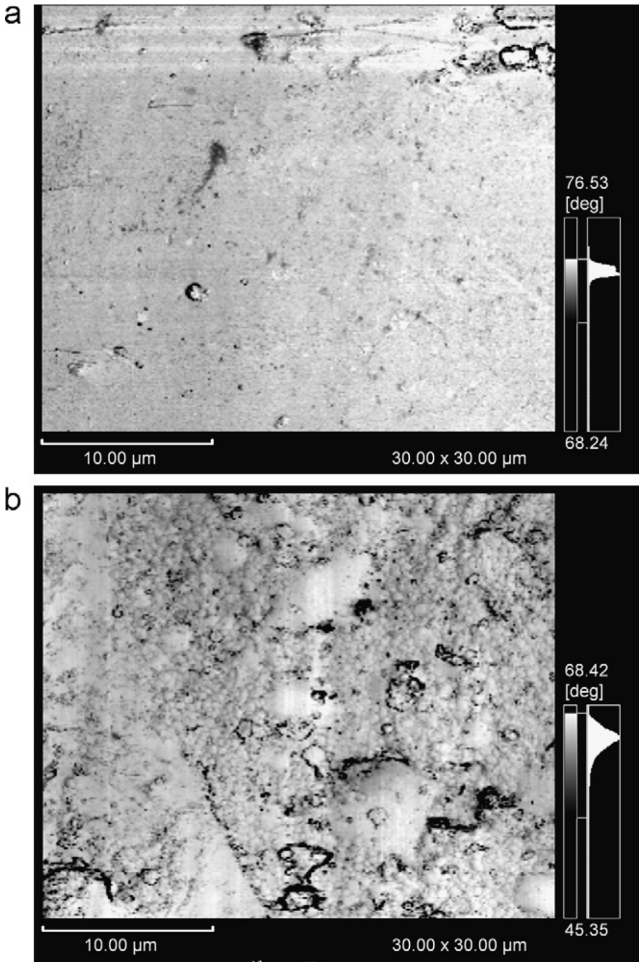

Fig. 5. SPM photographs of NZR to zirconia (a: surface of layering porcelain, b: fractured surface).

Table 5
Fracture mode of surface divided by numbers of specimen.

Materials	Surface treatments	Cohesive	Cohesive/ adhesive	Adhesive
NobelRondo Zirconia	#600	4	2	0
	Rocatec	5	1	0
Estenia C&B	#600	0	0	6
	Rocatec	0	0	6

caries of abutment and fracture are mentioned [40]. For ceramic frameworks, it is also demanded paying careful attention to a form and adjustment of frameworks, veneering the materials, and polishing. It is cleared that porcelain or hybrid composite material was loaded occlusal force repeatedly, and this will cause the abrasion of biting tooth or restorative material [41]. From this study, shear bond strength of NZR and ES to zirconia was recorded, however, it is expected that higher bond strength will need for restoration of hybrid composite to zirconia. When using in clinic, careful consideration about occlusal force, the region of restoration, the number of abutments, and the designing of restoration for each patient are important. Heikkinen et al. [42] reported that the effect of operating air pressure of tribochemical silica-coating method on the shear bond strength of composite resin to zirconia. And this study suggests that higher air pressures may have a significant effect on bond strength.

5. Conclusions

Within the limitation of this study, following conclusions are made:

1. Shear bond strength of layering porcelain to zirconia was significantly higher than that of restorative hybrid resin, however, there were no significant differences between surface treatments.
2. SPM photograph shows that cohesive fracture was observed in layering porcelain attached to zirconia surface.
3. More study will be needed, however, the bond strength of the hybrid composite resin to zirconia is not enough to use, appropriate choice of materials became the gides to the expansion of the applied cases of metal-free prothesis.

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